SPECIFICATION

NO DRAWINGS

1.056.331

e de la companya del companya de la companya del companya de la companya del la companya de la c



Date of Application and filing Complete Specification: Jan. 15, 1964.

Application made in Italy (No. 1104) on Jan. 18. 1963. Complete Specification Published: Jan. 25, 1967. © Crown Copyright 1967.

Index at acceptance:—C2 C(2B3A4, 2B3E, 2B3G1)

Int. Cl.:-C 07 d 7/04

COMPLETE SPECIFICATION

Process for Preparing Glucosamine Salts

Wc. ROTTA RESEARCH LABORATORIUM, an Italian Joint Stock Company, of San Fruttuoso di Monza, Milan, Italy, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:-

This invention relates to a process for

10 preparing glucosamine salts.

The invention provides a process for preparing glucosamine sulphate, phosphate or hydriodide comprising placing glucosamine hydrochloride solution in contact with an 15 anionic resin previously conditioned with sulphuric, phosphoric or hydriodic acid or a metal salt of one of these acids.

 $+2H^{\bullet}+SO_{\bullet}^{-}\longrightarrow R=SO_{\bullet}+2HCI$

By way of example, in order to obtain

glucosamine sulphate an anionic resin is

employed which may be represented by the

formula R.Cl., conditioned with an aqueous

solution of sulphate ions (sulphuric acid) in

accordance with the following reaction:

After washing the resin with distilled water, an exchange contact with a solution of glucosamine hydrochloride is carried out, giving rise to the following reaction:

Thie yields 110 gr white or slightly yellow-tinged crystals melting at 188—190° C. The

product in this case is glucosamine hydriodide.

EXAMPLE III The same procedure as described in Ex-

ample I is followed; however, the resin is

conditioned by means of an Na.HPO, solu-

water-soluble product melting at 195°

The result is a crystalline white highly

centesimal analysis discloses that the product

 $R=SO_4+2$ $C_4H_{11}O_5NH_7$. $HCI \rightarrow (C_6H_{11}O_5NH_2)_7$. $H_2SO_4+R.Cl_7$. ample I is followed; however, the resin is conditioned by means of a normal NaI solu-

Further details of the method will appear from the following Examples.

EXAMPLE I.

An anionic resin is conditioned in a column by means of a normal aqueous solution (1 N) of Na₂SO, at a rate of about 500 ml/hour. After washing the resin with distilled water, 1,400 ml of a 0.3 N glucosamine hydrochloride solution are led through the column at a rate of about 300 ml/hour.

The effluent solution from the column is collected and concentrated in vacuum at 45° -52° C to a volume of 200 ml, and is mixed with 200 ml acetone, whereupon the mixture is brought to dryness. The result is a crystal-line product which is washed with ethyl alcohol. About 90 gr white or slightly yellow-

tinged crystals are obtained, which melt at 115°-122° C, with decomposition at 127° C. Centesimal analysis discloses that the product is glucosamine sulphate.

WHAT WE CLAIM IS:-1. A process for preparing glucosamine

is glucosamine phosphate.

sulphate, phosphate or hydriodide, comprising placing glucosamine hydrochloride solution in contact with an anionic resin previously conditioned with sulphuric, phosphoric or hydriodic acid or a melt salt of one of these

EXAMPLE II The same procedure as described in Ex-

[Price 4s. 6d.]

BEST AVAILABLE COPY

- 2. A process as claimed in claim 1, substantially as nereinbefore described in Example I, II or III.
- 3. Glucosamine sulphate, phosphate or hydriodide when prepared by a process as claimed in claim 1 or claim 2.
- H. D. FITZPATRICK & CO., Chartered Patent Agents, 3, Grays Inn Square, London, W.C.1, and 5, Park Gardens, Glasgow, C.3.

Leamington Spa: Printed for Her Majesty's Stationery Office by the Courier Press.—1967.

Published at The Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.